

Hirshfeld atom refinement for modeling strong hydrogen bonds

(Supplementary Materials)

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Nomenclature scheme for all TAAM refinements:

- TAAM_synch - fully optimized geometry starting from the IAM synchrotron geometry;
- TAAM_0 - fully optimized geometry with H1 in the intramolecular hydrogen bond initially shifted to a symmetric position;
- TAAM_0_constr - optimized geometry with H1 in the intramolecular hydrogen bond initially shifted to a symmetric position and fixed H1-O1 distance;

- TAAM_005_constr, TAAM_010_constr, TAAM_015_constr (also called TAAM), TAAM_020_constr, TAAM_025_constr, TAAM_030_constr, TAAM_035_constr, TAAM_040_constr, TAAM_045_constr - optimized geometry with H1 in the intramolecular hydrogen bond initially shifted from the symmetric position by 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45 Å, respectively, and fixed H1-O1 distance.

Nomenclature scheme of the models chosen for geometry analysis:

- IAM - Independent Atom Model;
- neutron - neutron structure;
- TAAM (= TAAM_015_constr) - transferable aspherical atom model with the most symmetric intramolecular hydrogen bond after optimization;
- MM_iso - free multipole refinement with isotropically refined hydrogen atoms;
- MM_shade - free multipole refinement with hydrogen ADPs estimated by SHADE2 and fixed;
- HAR_iso - Hirshfeld atom refinement with hydrogen ADPs refined isotropically;
- HAR_aniso - Hirshfeld atom refinement with hydrogen ADPs refined anisotropically;
- HAR_shade - Hirshfeld atom refinement with hydrogen ADPs estimated by SHADE2 and fixed.

Table 1. Refinement details for all TAAMs

| | TAAM_synch | TAAM_0 | TAAM_0_constr | TAAM_005_constr |
|---|----------------------|----------------------|----------------------|----------------------|
| $R(F)$ | 0.0200 | 0.0206 | 0.0206 | 0.0206 |
| $R_w(F)$ | 0.0274 | 0.0282 | 0.0282 | 0.0280 |
| GOF | 0.91 | 0.92 | 0.92 | 0.92 |
| Condition for observed reflections | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ |
| No. of observed reflections | 7394 | 7394 | 7394 | 7394 |
| No. of parameters | 199 | 199 | 199 | 199 |
| $\Delta\varrho_{max}, \Delta\varrho_{min}$ ($e/\text{\AA}^3$) | 0.28, -0.25 | 0.28, -0.27 | 0.28, -0.27 | 0.29, -0.26 |

| | TAAM_010_constr | TAAM_015_constr | TAAM_020_constr | TAAM_025_constr |
|---|----------------------|----------------------|----------------------|----------------------|
| $R(F)$ | 0.0206 | 0.0283 | 0.0283 | 0.0284 |
| $R_w(F)$ | 0.0282 | 0.0210 | 0.0212 | 0.0211 |
| GOF | 0.92 | 0.92 | 0.92 | 0.93 |
| Condition for observed reflections | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ |
| No. of observed reflections | 7394 | 7394 | 7394 | 7394 |
| No. of parameters | 199 | 199 | 199 | 199 |
| $\Delta\varrho_{max}, \Delta\varrho_{min}$ ($e/\text{\AA}^3$) | 0.28, -0.27 | 0.28, -0.27 | 0.28, -0.27 | 0.28, -0.27 |

| | TAAM_030_constr | TAAM_035_constr | TAAM_040_constr | TAAM_045_constr |
|---|----------------------|----------------------|----------------------|----------------------|
| $R(F)$ | 0.0208 | 0.0209 | 0.0209 | 0.0210 |
| $R_w(F)$ | 0.0284 | 0.0285 | 0.0285 | 0.0285 |
| GOF | 0.93 | 0.93 | 0.93 | 0.93 |
| Condition for observed reflections | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ | $F^2 > 2\sigma(F^2)$ |
| No. of observed reflections | 7394 | 7394 | 7394 | 7394 |
| No. of parameters | 199 | 199 | 199 | 199 |
| $\Delta\varrho_{max}, \Delta\varrho_{min}$ ($e/\text{\AA}^3$) | 0.28, -0.27 | 0.28, -0.27 | 0.28, -0.28 | 0.28, -0.28 |

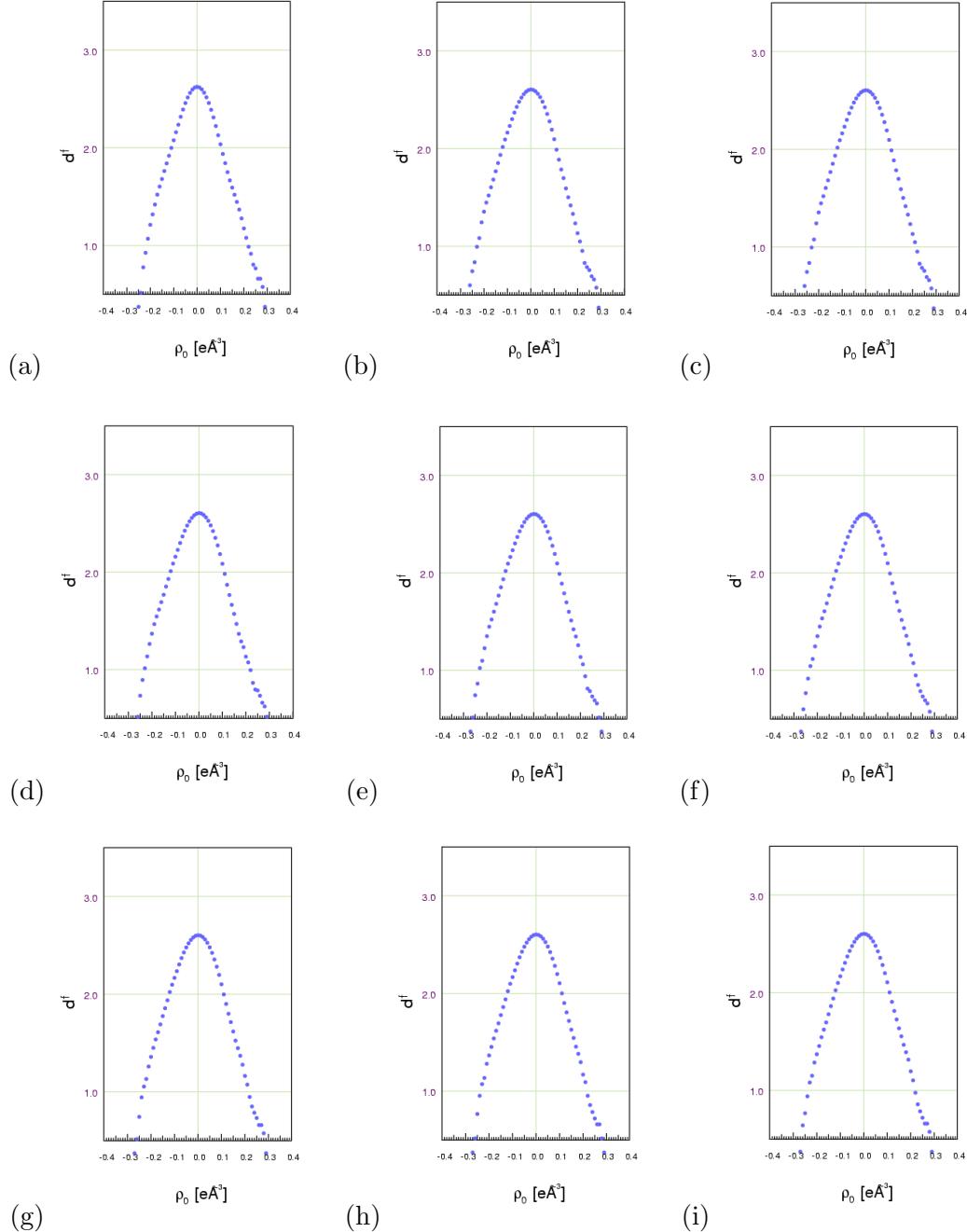


Fig. 1. Fractal dimension plots (fractal dimension d^f vs. residual density ρ_0) obtained for various TAAM refinements: (a) TAAM_synch, (b) TAAM_0, (c) TAAM_0_constr, (d) TAAM_005_constr, (e) TAAM_010_constr, (f) TAAM_015_constr (also called TAAM), (g) TAAM_020_constr, (h) TAAM_025_constr, (i) TAAM_030_constr.

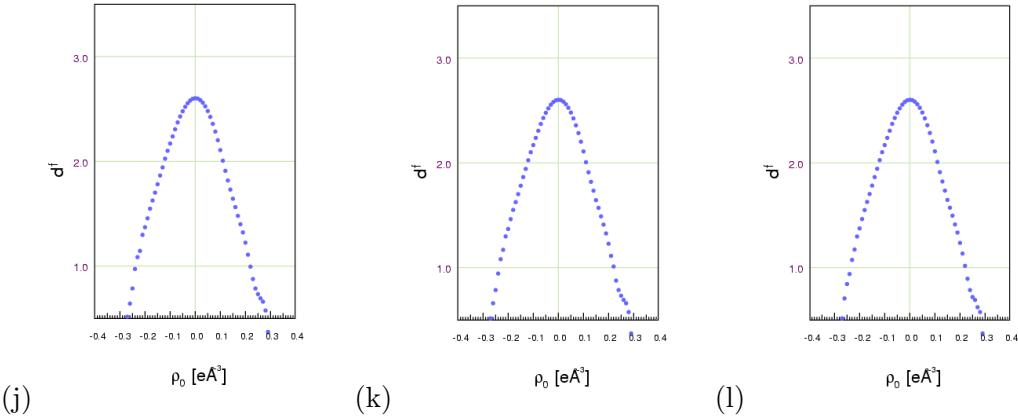


Fig. 2. Fractal dimension plots (fractal dimension d^f vs. residual density ρ_0) obtained for various TAAM refinements: (j) TAAM_035_constr, (k) TAAM_040_constr, (l) TAAM_045_constr.

Table 2. Geometry of the intramolecular hydrogen bond $O1 - H1 \cdots O2$ and selected torsion angles for the hydrogen maleate anion obtained with various TAAM refinements. Units of distances and angles: Å, deg.

| Model | O1 - H1 | H1···O2 | O1···O2 | O1 - H1···O2 | C1-C2-C3-C4 |
|------------------|-----------|-----------|-----------|--------------|-------------|
| TAAM_synch | 1.310(20) | 1.100(20) | 2.4110(3) | 178.9(18) | -2.0(3) |
| TAAM_0 | 1.135(17) | 1.277(17) | 2.4112(3) | 177.9(14) | -2.0(3) |
| TAAM_0..constr | 1.136(17) | 1.276(17) | 2.4112(3) | 177.9(14) | -2.0(3) |
| TAAM_005..constr | 1.236(19) | 1.175(19) | 2.4108(3) | 177.6(15) | -2.0(3) |
| TAAM_010..constr | 1.190(17) | 1.222(17) | 2.4111(3) | 178.2(14) | -2.0(3) |
| TAAM_015..constr | 1.218(17) | 1.194(16) | 2.4110(3) | 178.3(14) | -2.0(3) |
| TAAM_020..constr | 1.237(16) | 1.174(16) | 2.4108(3) | 178.0(15) | -2.0(3) |
| TAAM_025..constr | 1.248(15) | 1.164(15) | 2.4108(3) | 177.4(17) | -2.0(3) |
| TAAM_030..constr | 1.255(15) | 1.157(15) | 2.4108(3) | 176.7(19) | -2.0(3) |
| TAAM_035..constr | 1.256(15) | 1.156(14) | 2.4109(3) | 176.2(20) | -2.0(3) |
| TAAM_040..constr | 1.257(14) | 1.156(14) | 2.4111(3) | 175.8(21) | -2.0(3) |
| TAAM_045..constr | 1.259(14) | 1.154(14) | 2.4115(3) | 175.7(21) | -2.0(3) |

Table 3. $X - H$ distances (in Å) for hydrogen atoms (except H1 and H5) resulting from various refinement methods. The TAAM/MM distances are standard averaged values derived from neutron measurements.

| Method | C2-H2 maleate ring | C3-H3 maleate ring | N1-H41 ammonium group | N1-H42 ammonium group | N1-H43 ammonium group |
|-----------|-----------------------|-----------------------|--------------------------|--------------------------|--------------------------|
| IAM | 0.931(11) | 0.962(12) | 0.860(12) | 0.829(11) | 0.870(12) |
| neutron | 1.087(2) | 1.084(3) | 1.038(2) | 1.041(3) | 1.032(2) |
| TAAM/MM | 1.083 | 1.083 | 1.036 | 1.036 | 1.036 |
| HAR_iso | 1.065(11) | 1.083(11) | 1.024(14) | 0.992(12) | 1.040(10) |
| HAR_aniso | 1.050(10) | 1.077(11) | 1.037(13) | 0.999(13) | 1.022(10) |
| HAR_shade | 1.066(10) | 1.076(10) | 1.022(10) | 0.996(10) | 1.044(9) |

| Method | C6-H6 methine group | C7-H71 methylene group | C7-H72 methylene group | C9-H9 phenyl group | C10-H10 phenyl group |
|-----------|------------------------|---------------------------|---------------------------|-----------------------|-------------------------|
| IAM | 0.990(8) | 1.024(12) | 1.008(9) | 0.953(12) | 1.039(12) |
| neutron | 1.092(2) | 1.096(3) | 1.100(3) | 1.087(3) | 1.084(3) |
| TAAM/MM | 1.098 | 1.092 | 1.092 | 1.083 | 1.083 |
| HAR_iso | 1.102(9) | 1.086(10) | 1.083(9) | 1.077(12) | 1.123(10) |
| HAR_aniso | 1.100(10) | 1.075(10) | 1.076(10) | 1.068(11) | 1.087(10) |
| HAR_shade | 1.101(9) | 1.089(10) | 1.082(8) | 1.085(9) | 1.114(8) |

| Method | C11-H11 phenyl group | C12-H12 phenyl group | C13-H13 phenyl group | | |
|-----------|-------------------------|-------------------------|-------------------------|--|--|
| IAM | 0.991(11) | 0.979(11) | 0.915(11) | | |
| neutron | 1.088(3) | 1.088(3) | 1.091(3) | | |
| TAAM/MM | 1.083 | 1.083 | 1.083 | | |
| HAR_iso | 1.098(11) | 1.097(12) | 1.085(10) | | |
| HAR_aniso | 1.099(10) | 1.086(10) | 1.082(9) | | |
| HAR_shade | 1.096(10) | 1.095(9) | 1.084(8) | | |

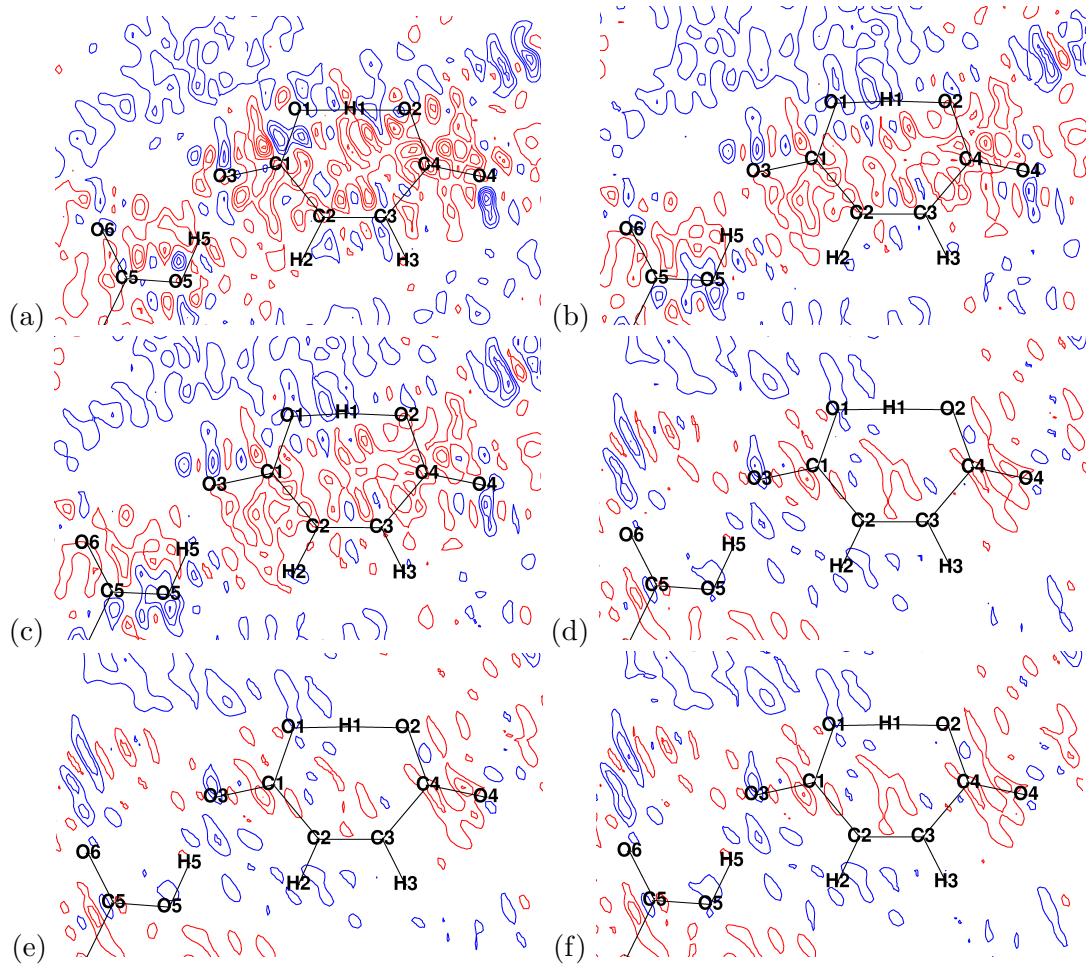


Fig. 3. Residual density maps in the plane of the ring of the maleate anion obtained after the following refinement procedures: (a) TAAM, (b) MM_iso, (c) MM_shade, (d) HAR_iso, (e) HAR_aniso, (f) HAR_shade. Contour level 0.05 e/ \AA^3 , blue - positive, red - negative.

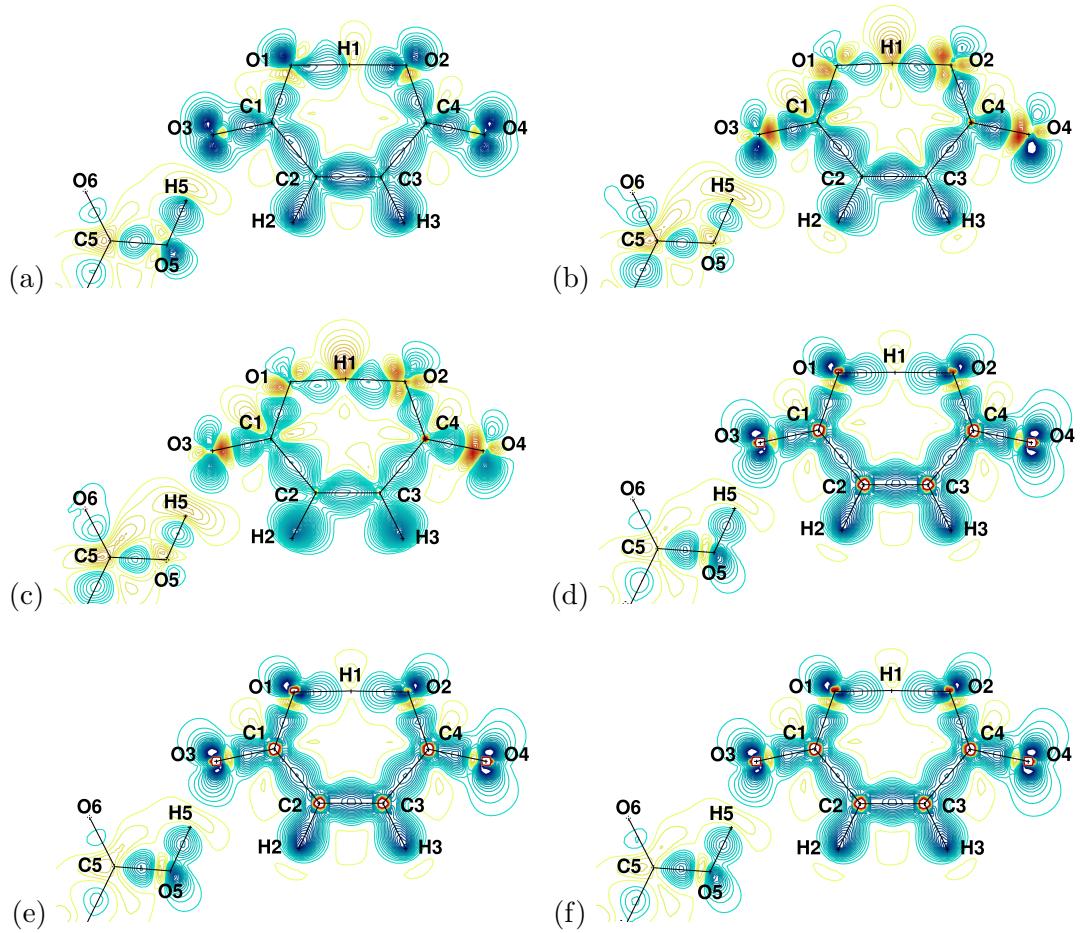


Fig. 4. Deformation density maps in the plane of the ring of the maleate anion obtained after the following refinement procedures: (a) TAAM, (b) MM_iso, (c) MM_shade, (d) HAR_iso, (e) HAR_aniso, (f) HAR_shade. Contour level 0.05 e/ \AA^3 , blue - positive, red - negative.

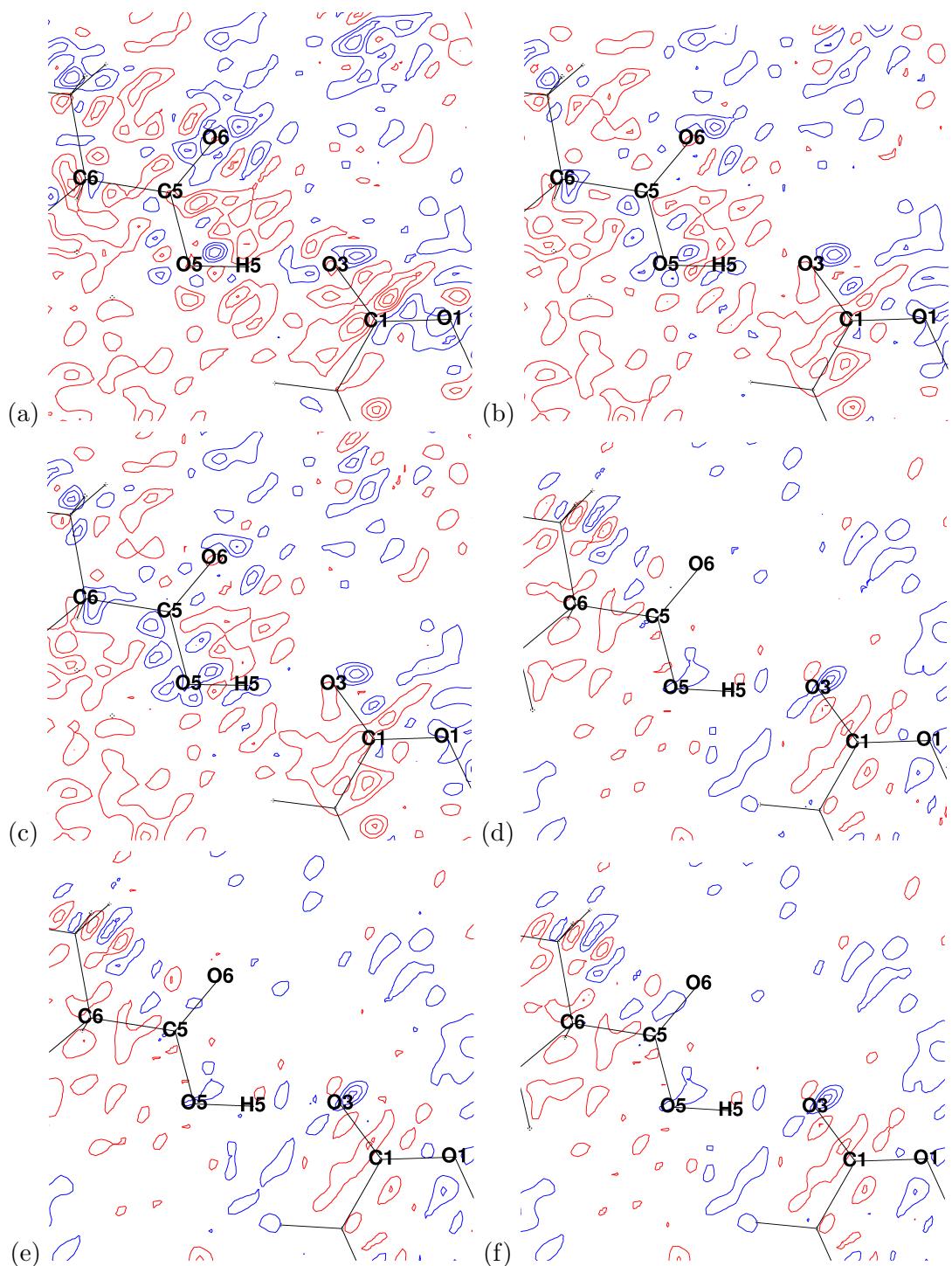


Fig. 5. Residual density maps depicting the $O_5 - H_5 \cdots O_3$ hydrogen bond (in the plane defined by atoms O_3 , O_5 and O_6) after the following refinement procedures: (a) TAAM, (b) MM_iso, (c) MM_shade, (d) HAR_iso, (e) HAR_aniso, (f) HAR_shade. Contour level $0.05 \text{ e}/\text{\AA}^3$, blue - positive, red - negative.

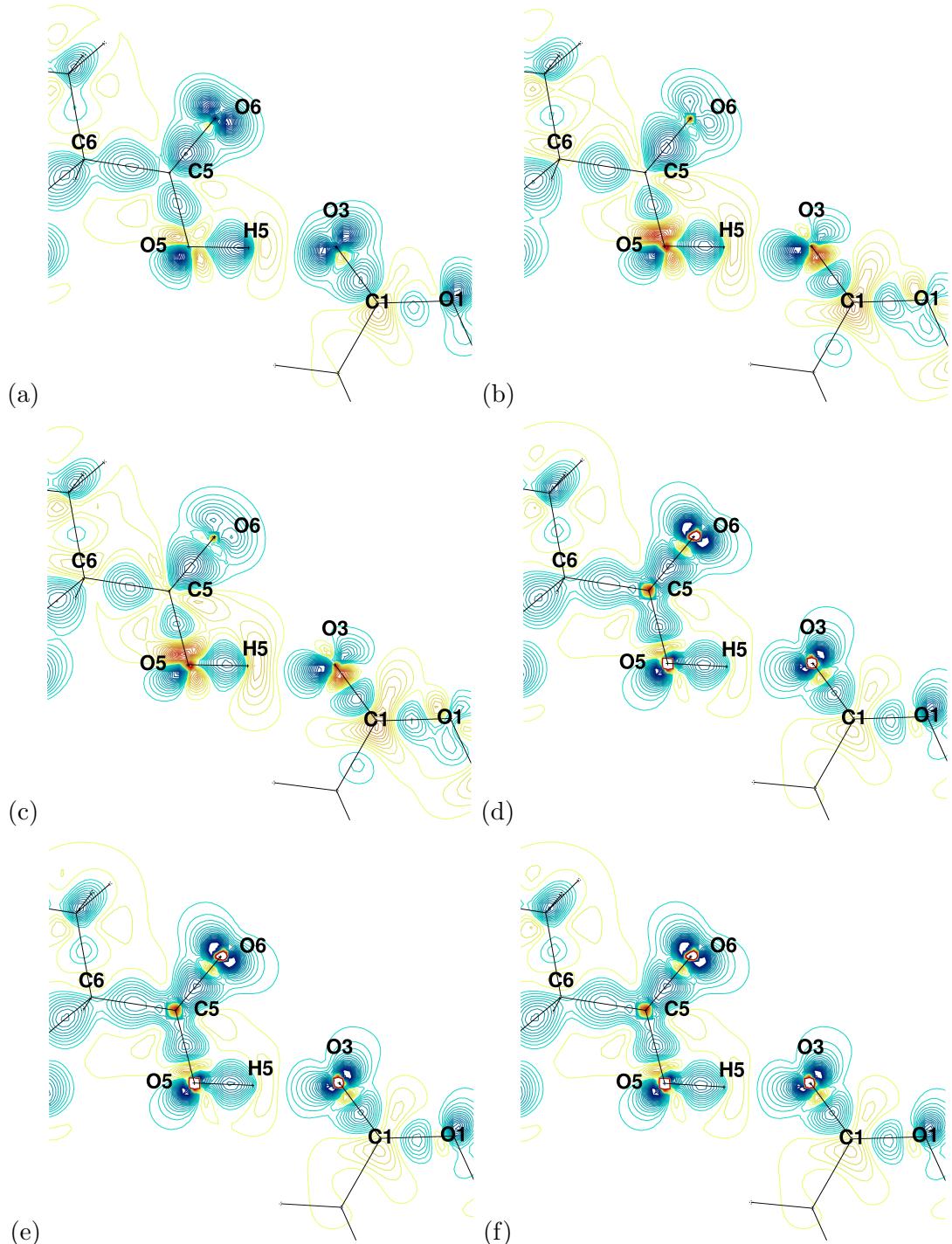


Fig. 6. Deformation density maps depicting the $\text{O}5 - \text{H}5 \cdots \text{O}3$ hydrogen bond (in the plane defined by atoms $\text{O}3$, $\text{O}5$ and $\text{O}6$) after the following refinement procedures: (a) TAAM, (b) MM_iso, (c) MM_shade, (d) HAR_iso, (e) HAR_aniso, (f) HAR_shade. Contour level $0.05 \text{ e}/\text{\AA}^3$, blue - positive, red - negative.

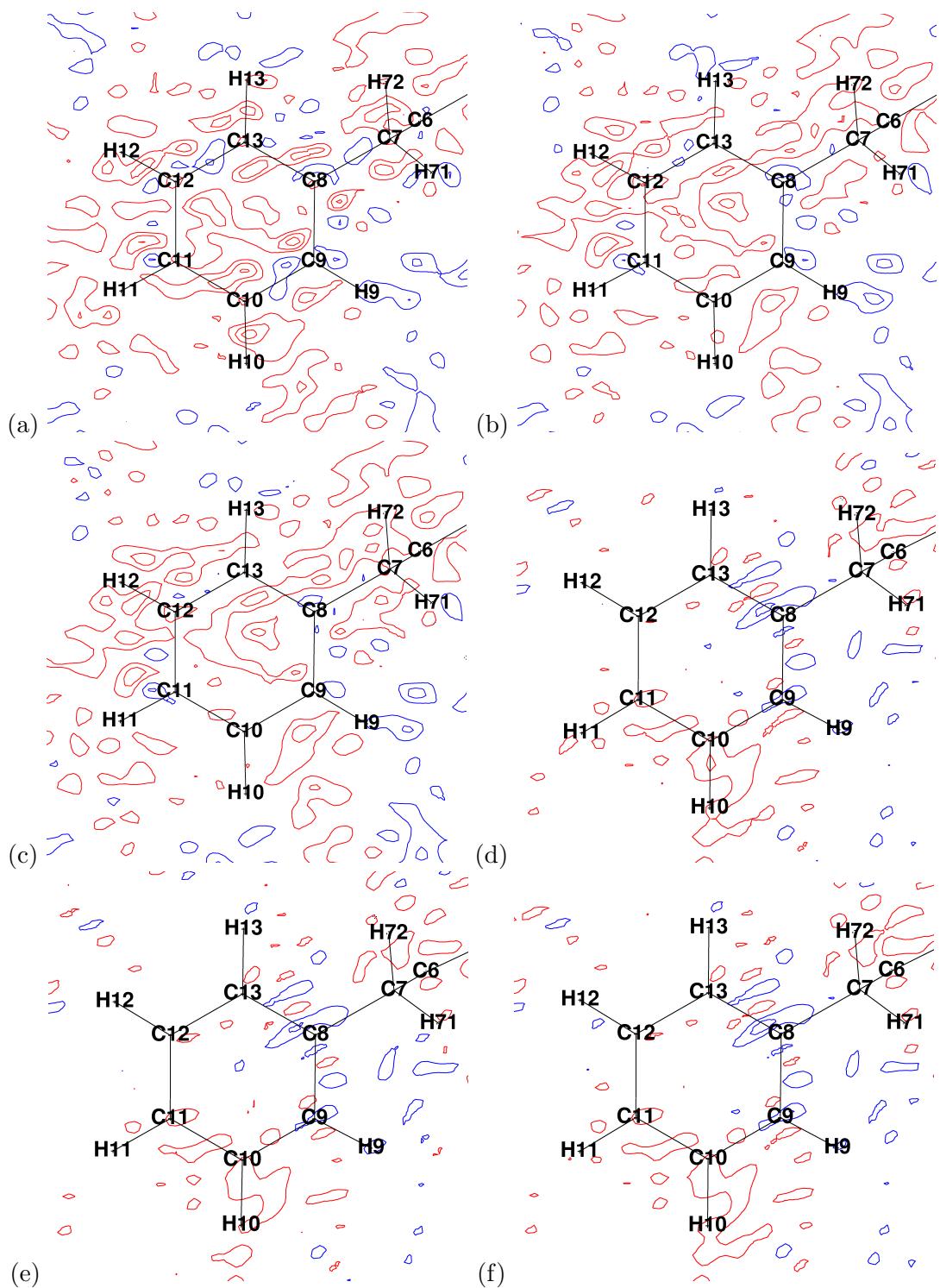


Fig. 7. Residual density maps in the plane of the phenyl ring obtained after the following refinement procedures: (a) TAAM, (b) MM_iso, (c) MM_shade, (d) HAR_iso, (e) HAR_aniso, (f) HAR_shade. Contour level 0.05 e/Å³, blue - positive, red - negative.

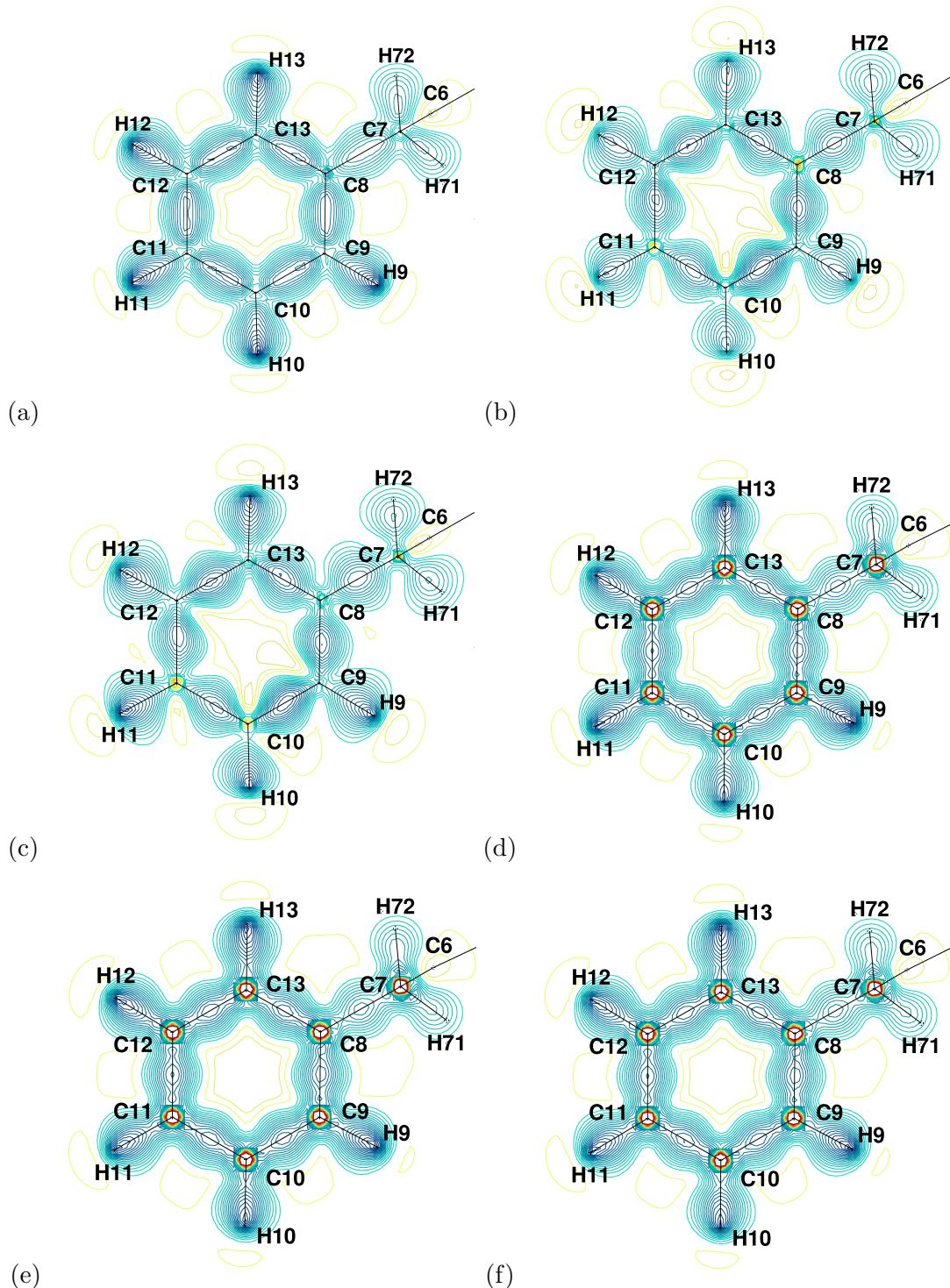


Fig. 8. Deformation density maps in the plane of the phenyl ring obtained after the following refinement procedures: (a) TAAM, (b) MM_iso, (c) MM_shade, (d) HAR_iso, (e) HAR_aniso, (f) HAR_shade. Contour level 0.05 e/Å³, blue - positive, red - negative.

Table 4. *Values of ADPs of hydrogen atoms for various refinement strategies (neutron, HAR_aniso, HAR_shade and MM_shade). The ADPs are expressed in the crystal coordinate system.*

| | Model | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|-----|-----------|------------|------------|-------------|-------------|-------------|-------------|
| H1 | neutron | 0.0220(10) | 0.0150(8) | 0.0229(10) | -0.0033(7) | 0.0043(9) | -0.0007(7) |
| | HAR_aniso | 0.0226(54) | 0.0095(51) | 0.0530(120) | -0.0088(42) | 0.0151(62) | -0.0064(61) |
| | HAR_shade | 0.0323 | 0.0216 | 0.0142 | -0.0046 | 0.0074 | -0.0029 |
| | MM_shade | 0.0328 | 0.0218 | 0.0154 | -0.0043 | 0.0083 | -0.0032 |
| H2 | neutron | 0.0375(14) | 0.0145(8) | 0.0142(8) | -0.0080(9) | 0.0070(9) | -0.0063(7) |
| | HAR_aniso | 0.0473(52) | 0.0226(48) | 0.0164(41) | -0.0100(41) | 0.0107(38) | -0.0022(36) |
| | HAR_shade | 0.0503 | 0.0206 | 0.0205 | -0.0121 | 0.0077 | -0.0084 |
| | MM_shade | 0.0503 | 0.0209 | 0.0208 | -0.0109 | 0.0079 | -0.0084 |
| H3 | neutron | 0.0347(13) | 0.0136(8) | 0.0181(9) | -0.0091(8) | 0.0061(10) | 0.0017(7) |
| | HAR_aniso | 0.0411(47) | 0.0287(50) | 0.0104(38) | -0.0074(42) | 0.0148(36) | -0.0040(37) |
| | HAR_shade | 0.0496 | 0.0208 | 0.0233 | -0.0128 | 0.0104 | 0.0007 |
| | MM_shade | 0.0485 | 0.0209 | 0.0240 | -0.0122 | 0.0103 | 0.0008 |
| H41 | neutron | 0.0233(11) | 0.0191(9) | 0.0201(9) | -0.0018(8) | 0.0058(9) | -0.0107(8) |
| | HAR_aniso | 0.0229(43) | 0.0352(63) | 0.0352(61) | -0.0095(44) | 0.0167(41) | -0.0155(54) |
| | HAR_shade | 0.0259 | 0.0219 | 0.0228 | 0.0007 | 0.0045 | -0.0111 |
| | MM_shade | 0.0260 | 0.0219 | 0.0237 | 0.0000 | 0.0043 | -0.0109 |
| H42 | neutron | 0.0258(11) | 0.0214(9) | 0.0172(9) | 0.0057(8) | 0.0048(9) | 0.0057(8) |
| | HAR_aniso | 0.0277(43) | 0.0357(62) | 0.0245(54) | 0.0018(42) | 0.0164(39) | -0.0102(48) |
| | HAR_shade | 0.0236 | 0.0222 | 0.0190 | 0.0052 | 0.0053 | 0.0041 |
| | MM_shade | 0.0236 | 0.0230 | 0.0197 | 0.0056 | 0.0051 | 0.0040 |
| H43 | neutron | 0.0108(8) | 0.0277(11) | 0.0226(10) | -0.0031(7) | 0.0061(8) | -0.0034(8) |
| | HAR_aniso | 0.0179(36) | 0.0394(66) | 0.0300(56) | -0.0076(41) | 0.0154(36) | -0.0009(53) |
| | HAR_shade | 0.0127 | 0.0314 | 0.0222 | -0.0022 | 0.0026 | -0.0032 |
| | MM_shade | 0.0133 | 0.0315 | 0.0232 | -0.0028 | 0.0029 | -0.0034 |
| H5 | neutron | 0.0199(9) | 0.0188(9) | 0.0185(9) | -0.0025(8) | 0.0081(8) | -0.0045(8) |
| | HAR_aniso | 0.0277(61) | 0.0261(72) | 0.0408(76) | -0.0074(58) | -0.0044(53) | 0.0085(63) |
| | HAR_shade | 0.0265 | 0.0259 | 0.0187 | -0.0055 | 0.0066 | -0.0084 |
| | MM_shade | 0.0269 | 0.0259 | 0.0193 | -0.0060 | 0.0066 | -0.0081 |
| H6 | neutron | 0.0146(8) | 0.0139(8) | 0.0180(9) | 0.0039(6) | 0.0009(8) | 0.0035(7) |
| | HAR_aniso | 0.0197(34) | 0.0298(50) | 0.0074(32) | 0.0009(35) | 0.0048(27) | 0.0045(33) |
| | HAR_shade | 0.0199 | 0.0188 | 0.0232 | 0.0029 | -0.0011 | 0.0032 |
| | MM_shade | 0.0212 | 0.0191 | 0.0231 | 0.0019 | -0.0010 | 0.0038 |
| H71 | neutron | 0.0206(10) | 0.0124(8) | 0.0248(10) | 0.0006(7) | -0.0002(9) | -0.0037(8) |
| | HAR_aniso | 0.0401(58) | 0.0143(42) | 0.0343(49) | 0.0011(44) | -0.0174(42) | -0.0091(39) |
| | HAR_shade | 0.0255 | 0.0183 | 0.0328 | 0.0011 | -0.0018 | -0.0081 |
| | MM_shade | 0.0256 | 0.0195 | 0.0328 | 0.0010 | -0.0016 | -0.0086 |
| H72 | neutron | 0.0178(9) | 0.0256(11) | 0.0158(9) | -0.0006(8) | 0.0072(8) | 0.0055(8) |
| | HAR_aniso | 0.0383(51) | 0.0196(46) | 0.0228(43) | -0.0069(41) | -0.0014(37) | 0.0078(37) |
| | HAR_shade | 0.0229 | 0.0356 | 0.0189 | -0.0024 | 0.0054 | 0.0048 |
| | MM_shade | 0.0234 | 0.0362 | 0.0195 | -0.0026 | 0.0053 | 0.0049 |

Table 5. *Values of ADPs of hydrogen atoms for various refinement strategies (neutron, HAR_aniso, HAR_shade and MM_shade). The ADPs are expressed in the crystal coordinate system.*

| | Model | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|-----|-----------|------------|------------|------------|------------|-------------|-------------|
| H9 | neutron | 0.0259(11) | 0.0241(10) | 0.0181(9) | 0.0104(9) | 0.0058(9) | -0.0040(8) |
| | HAR_aniso | 0.0344(56) | 0.0335(60) | 0.0378(54) | 0.0186(50) | -0.0049(44) | -0.0146(49) |
| | HAR_shade | 0.0283 | 0.0280 | 0.0231 | 0.0105 | 0.0035 | -0.0080 |
| | MM_shade | 0.0284 | 0.0301 | 0.0231 | 0.0106 | 0.0045 | -0.0076 |
| H10 | neutron | 0.0320(13) | 0.0326(13) | 0.0111(8) | 0.0103(10) | 0.0022(9) | -0.0010(8) |
| | HAR_aniso | 0.0432(57) | 0.0422(67) | 0.0173(47) | 0.0242(51) | 0.0095(40) | 0.0009(47) |
| | HAR_shade | 0.0329 | 0.0419 | 0.0130 | 0.0073 | 0.0005 | -0.0017 |
| | MM_shade | 0.0340 | 0.0416 | 0.0139 | 0.0075 | 0.0013 | -0.0017 |
| H11 | neutron | 0.0253(11) | 0.0282(11) | 0.0206(10) | 0.0140(10) | 0.0021(9) | 0.0055(9) |
| | HAR_aniso | 0.0303(41) | 0.0290(53) | 0.0227(49) | 0.0103(39) | 0.0168(36) | 0.0119(42) |
| | HAR_shade | 0.0294 | 0.0329 | 0.0263 | 0.0133 | -0.0010 | 0.0074 |
| | MM_shade | 0.0305 | 0.0325 | 0.0274 | 0.0134 | -0.0007 | 0.0072 |
| H12 | neutron | 0.0284(12) | 0.0248(11) | 0.0201(10) | 0.0122(9) | 0.0066(10) | -0.0036(8) |
| | HAR_aniso | 0.0237(38) | 0.0390(65) | 0.0326(57) | 0.0110(41) | 0.0191(37) | -0.0019(50) |
| | HAR_shade | 0.0296 | 0.0309 | 0.0300 | 0.0141 | 0.0083 | -0.0047 |
| | MM_shade | 0.0295 | 0.0318 | 0.0311 | 0.0142 | 0.0087 | -0.0041 |
| H13 | neutron | 0.0258(12) | 0.0322(12) | 0.0070(7) | 0.0086(9) | 0.0027(8) | 0.0007(7) |
| | HAR_aniso | 0.0281(39) | 0.0474(66) | 0.0091(35) | 0.0134(44) | 0.0119(31) | 0.0006(41) |
| | HAR_shade | 0.0290 | 0.0345 | 0.0140 | 0.0054 | 0.0059 | -0.0021 |
| | MM_shade | 0.0294 | 0.0352 | 0.0148 | 0.0052 | 0.0062 | -0.0017 |

Table 6. Values of ADPs of non-hydrogen atoms for various refinement strategies (IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_shade and HAR_aniso). The ADPs are expressed in the crystal coordinate system.

| Model | | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|-------|-------------------|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | IAM | 0.01644(11) | 0.01107(9) | 0.00887(9) | -0.00370(8) | 0.00371(8) | -0.00271(7) |
| | neutron | 0.00870(40) | 0.00630(40) | 0.00400(40) | -0.00180(30) | 0.00200(30) | -0.00130(30) |
| | TAAM | 0.01628(8) | 0.01076(7) | 0.00895(7) | -0.00347(6) | 0.00368(5) | -0.00248(5) |
| | MM_iso | 0.01640(10) | 0.01079(8) | 0.00935(10) | -0.00346(8) | 0.00357(7) | -0.00249(7) |
| | MM_shade | 0.01638(10) | 0.01078(8) | 0.00936(10) | -0.00347(8) | 0.00357(7) | -0.00249(7) |
| | HAR_iso/HAR_shade | 0.01646(7) | 0.01068(7) | 0.00853(7) | -0.00359(6) | 0.00372(6) | -0.00249(5) |
| | HAR_aniso | 0.01648(7) | 0.01068(7) | 0.00850(7) | -0.00356(6) | 0.00370(6) | -0.00250(5) |
| O2 | IAM | 0.01933(13) | 0.01046(10) | 0.00892(9) | -0.00166(9) | 0.00335(8) | -0.00215(8) |
| | neutron | 0.01140(40) | 0.00570(30) | 0.00530(40) | -0.00170(30) | 0.00200(40) | -0.00160(30) |
| | TAAM | 0.01948(8) | 0.01051(7) | 0.00813(7) | -0.00152(6) | 0.00377(6) | -0.00261(5) |
| | MM_iso | 0.01933(12) | 0.01037(9) | 0.00896(10) | -0.00159(7) | 0.00376(6) | -0.00239(8) |
| | MM_shade | 0.01933(12) | 0.01036(9) | 0.00895(10) | -0.00159(7) | 0.00374(6) | -0.00240(8) |
| | HAR_iso/HAR_shade | 0.01926(8) | 0.01050(7) | 0.00815(7) | -0.00152(6) | 0.00366(6) | -0.00258(6) |
| | HAR_aniso | 0.01927(8) | 0.01052(7) | 0.00808(7) | -0.00150(6) | 0.00365(6) | -0.00259(6) |
| O3 | IAM | 0.01484(11) | 0.01326(10) | 0.00762(9) | -0.00181(8) | 0.00411(7) | -0.00231(8) |
| | neutron | 0.00900(40) | 0.00780(40) | 0.00420(40) | -0.00150(30) | 0.00140(40) | -0.00150(30) |
| | TAAM | 0.01448(7) | 0.01241(7) | 0.00740(6) | -0.00227(6) | 0.00363(5) | -0.00226(5) |
| | HAR_iso/HAR_shade | 0.01431(10) | 0.01274(9) | 0.00797(10) | -0.00194(7) | 0.00352(6) | -0.00231(8) |
| | MM_shade | 0.01430(10) | 0.01271(9) | 0.00795(10) | -0.00194(7) | 0.00351(6) | -0.00231(8) |
| | HAR_iso | 0.01414(7) | 0.01251(7) | 0.00739(7) | -0.00203(5) | 0.00355(5) | -0.00217(5) |
| | HAR_aniso | 0.01417(7) | 0.01250(8) | 0.00736(7) | -0.00203(6) | 0.00354(5) | -0.00217(6) |
| O4 | IAM | 0.01607(11) | 0.01528(11) | 0.00752(9) | 0.00093(9) | 0.00416(8) | -0.00034(8) |
| | neutron | 0.01110(40) | 0.00720(40) | 0.00480(40) | -0.00070(30) | 0.00190(30) | -0.00010(30) |
| | TAAM | 0.01602(8) | 0.01445(8) | 0.00750(6) | 0.00027(6) | 0.00409(5) | -0.00080(6) |
| | MM_iso | 0.01592(11) | 0.01459(11) | 0.00809(10) | 0.00039(8) | 0.00410(6) | -0.00045(9) |
| | MM_shade | 0.01591(11) | 0.01461(11) | 0.00806(10) | 0.00040(8) | 0.00410(6) | -0.00045(9) |
| | HAR_iso/HAR_shade | 0.01598(7) | 0.01452(8) | 0.00723(7) | 0.00031(6) | 0.00405(6) | -0.00077(6) |
| | HAR_aniso | 0.01597(7) | 0.01455(8) | 0.00723(7) | 0.00034(6) | 0.00404(6) | -0.00075(6) |
| O5 | IAM | 0.01414(10) | 0.01495(10) | 0.00847(9) | -0.00444(9) | 0.00531(8) | -0.00339(8) |
| | neutron | 0.00900(40) | 0.00860(40) | 0.00540(40) | -0.00310(30) | 0.00220(40) | -0.00150(30) |
| | TAAM | 0.01375(7) | 0.01434(8) | 0.00871(6) | -0.00423(6) | 0.00509(5) | -0.00275(6) |
| | MM_iso | 0.01407(8) | 0.01445(8) | 0.00897(8) | -0.00444(9) | 0.00508(7) | -0.00300(8) |
| | MM_shade | 0.01407(8) | 0.01444(8) | 0.00896(8) | -0.00443(9) | 0.00509(7) | -0.00299(8) |
| | HAR_iso/HAR_shade | 0.01394(7) | 0.01418(8) | 0.00831(7) | -0.00443(6) | 0.00508(5) | -0.00296(6) |
| | HAR_aniso | 0.01392(7) | 0.01415(8) | 0.00832(7) | -0.00441(6) | 0.00503(5) | -0.00297(6) |
| O6 | IAM | 0.01031(8) | 0.01008(8) | 0.00963(9) | -0.00194(7) | 0.00290(6) | -0.00123(8) |
| | neutron | 0.00700(40) | 0.00630(30) | 0.00650(40) | -0.00250(30) | 0.00150(30) | -0.00140(30) |
| | TAAM | 0.01051(6) | 0.00956(6) | 0.00904(6) | -0.00203(5) | 0.00328(5) | -0.00132(5) |
| | MM_iso | 0.01053(7) | 0.00987(6) | 0.00931(7) | -0.00215(7) | 0.00311(6) | -0.00097(6) |
| | MM_shade | 0.01046(7) | 0.00979(6) | 0.00944(7) | -0.00207(7) | 0.00319(6) | -0.00099(6) |
| | HAR_iso/HAR_shade | 0.01046(6) | 0.00952(6) | 0.00873(7) | -0.00194(5) | 0.00323(5) | -0.00132(5) |
| | HAR_aniso | 0.01045(6) | 0.00950(6) | 0.00876(7) | -0.00193(5) | 0.00326(5) | -0.00132(5) |

Table 7. Values of ADPs of non-hydrogen atoms for various refinement strategies (IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_shade and HAR_aniso). The ADPs are expressed in the crystal coordinate system.

| | Model | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|----|-------------------|-------------|-------------|-------------|--------------|-------------|--------------|
| N1 | IAM | 0.00928(9) | 0.01343(11) | 0.00934(10) | -0.00005(8) | 0.00226(8) | -0.00335(9) |
| | neutron | 0.00500(20) | 0.00790(20) | 0.00520(20) | -0.00027(19) | 0.00130(20) | -0.00171(19) |
| | TAAM | 0.00905(6) | 0.01322(8) | 0.00900(7) | 0.00004(6) | 0.00232(5) | -0.00337(6) |
| | MM_iso | 0.00934(6) | 0.01338(8) | 0.00929(7) | 0.00006(5) | 0.00229(6) | -0.00337(5) |
| | MM_shade | 0.00932(6) | 0.01334(8) | 0.00929(7) | 0.00008(5) | 0.00228(6) | -0.00337(5) |
| | HAR_iso/HAR_shade | 0.00898(6) | 0.01308(8) | 0.00862(7) | 0.00013(6) | 0.00222(5) | -0.00330(6) |
| | HAR_aniso | 0.00897(6) | 0.01308(8) | 0.00862(8) | 0.00012(6) | 0.00224(5) | -0.00327(6) |
| C1 | IAM | 0.00953(11) | 0.01008(11) | 0.00801(11) | -0.00020(8) | 0.00192(8) | -0.00187(9) |
| | neutron | 0.00590(30) | 0.00470(30) | 0.00430(30) | -0.00030(20) | 0.00170(30) | -0.00080(20) |
| | TAAM | 0.00997(7) | 0.01011(8) | 0.00680(7) | -0.00058(6) | 0.00216(5) | -0.00175(6) |
| | MM_iso | 0.01033(7) | 0.01029(8) | 0.00732(8) | -0.00064(6) | 0.00220(5) | -0.00184(6) |
| | MM_shade | 0.01033(7) | 0.01025(8) | 0.00729(8) | -0.00063(6) | 0.00219(5) | -0.00183(6) |
| | HAR_iso/HAR_shade | 0.00959(7) | 0.00972(8) | 0.00675(8) | -0.00052(6) | 0.00211(6) | -0.00174(6) |
| | HAR_aniso | 0.00956(7) | 0.00974(8) | 0.00674(8) | -0.00051(6) | 0.00210(6) | -0.00176(6) |
| C2 | IAM | 0.01319(12) | 0.00993(11) | 0.00808(10) | -0.00151(9) | 0.00165(8) | -0.00164(9) |
| | neutron | 0.00770(30) | 0.00540(30) | 0.00540(30) | -0.00100(30) | 0.00200(30) | -0.00060(30) |
| | TAAM | 0.01362(8) | 0.00994(8) | 0.00703(8) | -0.00157(6) | 0.00213(6) | -0.00144(6) |
| | MM_iso | 0.01398(9) | 0.01021(8) | 0.00756(9) | -0.00169(7) | 0.00223(6) | -0.00143(7) |
| | MM_shade | 0.01395(9) | 0.01022(8) | 0.00752(9) | -0.00166(7) | 0.00224(6) | -0.00138(7) |
| | HAR_iso/HAR_shade | 0.01340(8) | 0.00952(8) | 0.00690(8) | -0.00159(6) | 0.00205(6) | -0.00159(6) |
| | HAR_aniso | 0.01341(8) | 0.00950(8) | 0.00685(8) | -0.00163(6) | 0.00206(6) | -0.00158(6) |
| C3 | IAM | 0.01411(12) | 0.01064(11) | 0.00776(10) | -0.00120(10) | 0.00242(9) | -0.00108(9) |
| | neutron | 0.00930(40) | 0.00480(30) | 0.00470(30) | -0.00180(30) | 0.00170(30) | -0.00060(30) |
| | TAAM | 0.01412(8) | 0.01025(8) | 0.00739(7) | -0.00143(7) | 0.00242(6) | -0.00109(6) |
| | MM_iso | 0.01432(9) | 0.01061(9) | 0.00787(8) | -0.00134(7) | 0.00245(6) | -0.00114(7) |
| | MM_shade | 0.01427(9) | 0.01064(9) | 0.00778(8) | -0.00131(7) | 0.00242(6) | -0.00118(7) |
| | HAR_iso/HAR_shade | 0.01380(8) | 0.01006(8) | 0.00713(8) | -0.00141(7) | 0.00237(6) | -0.00117(6) |
| | HAR_aniso | 0.01383(8) | 0.01002(8) | 0.00707(8) | -0.00144(7) | 0.00236(6) | -0.00117(7) |
| C4 | IAM | 0.01109(11) | 0.01131(11) | 0.00743(10) | 0.00111(9) | 0.00198(8) | -0.00142(9) |
| | neutron | 0.00600(30) | 0.00620(30) | 0.00360(30) | 0.00020(30) | 0.00180(30) | -0.00040(30) |
| | TAAM | 0.01138(8) | 0.01091(8) | 0.00680(7) | 0.00060(6) | 0.00231(6) | -0.00128(6) |
| | MM_iso | 0.01169(8) | 0.01125(9) | 0.00730(8) | 0.00065(6) | 0.00235(6) | -0.00129(6) |
| | MM_shade | 0.01168(8) | 0.01123(9) | 0.00726(8) | 0.00064(6) | 0.00234(6) | -0.00130(6) |
| | HAR_iso/HAR_shade | 0.01101(7) | 0.01069(8) | 0.00660(8) | 0.00068(6) | 0.00212(6) | -0.00140(6) |
| | HAR_aniso | 0.01096(7) | 0.01070(8) | 0.00664(8) | 0.00067(6) | 0.00215(6) | -0.00140(6) |
| C5 | IAM | 0.00786(9) | 0.00935(10) | 0.00679(9) | -0.00034(8) | 0.00141(7) | -0.00086(8) |
| | neutron | 0.00490(30) | 0.00440(30) | 0.00380(30) | -0.00050(20) | 0.00120(30) | -0.00020(20) |
| | TAAM | 0.00794(7) | 0.00892(7) | 0.00667(7) | -0.00072(6) | 0.00142(5) | -0.00078(6) |
| | MM_iso | 0.00825(6) | 0.00929(7) | 0.00696(7) | -0.00078(6) | 0.00151(5) | -0.00077(6) |
| | MM_shade | 0.00822(6) | 0.00927(7) | 0.00695(7) | -0.00078(6) | 0.00150(5) | -0.00077(6) |
| | HAR_iso/HAR_shade | 0.00767(6) | 0.00881(7) | 0.00615(7) | -0.00059(6) | 0.00126(5) | -0.00076(6) |
| | HAR_aniso | 0.00769(7) | 0.00879(7) | 0.00611(7) | -0.00060(6) | 0.00126(5) | -0.00077(6) |

Table 8. Values of ADPs of non-hydrogen atoms for various refinement strategies (IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_shade and HAR_aniso). The ADPs are expressed in the crystal coordinate system.

| | Model | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|-----|-------------------|-------------|-------------|-------------|--------------|-------------|--------------|
| C6 | IAM | 0.00807(10) | 0.00908(10) | 0.00771(10) | 0.00018(8) | 0.00069(8) | -0.00087(8) |
| | neutron | 0.00400(30) | 0.00440(30) | 0.00360(30) | -0.00020(20) | 0.00020(30) | 0.00040(20) |
| | TAAM | 0.00779(7) | 0.00868(7) | 0.00814(7) | 0.00009(6) | 0.00037(5) | -0.00092(6) |
| | MM_iso | 0.00805(7) | 0.00914(8) | 0.00846(8) | 0.00013(6) | 0.00050(6) | -0.00084(7) |
| | MM_shade | 0.00802(7) | 0.00911(8) | 0.00845(8) | 0.00013(6) | 0.00050(6) | -0.00085(7) |
| | HAR_iso/HAR_shade | 0.00766(7) | 0.00837(7) | 0.00778(8) | 0.00008(6) | 0.00032(5) | -0.00099(6) |
| | HAR_aniso | 0.00764(7) | 0.00840(8) | 0.00775(8) | 0.00008(6) | 0.00033(6) | -0.00101(6) |
| C7 | IAM | 0.00882(10) | 0.00960(10) | 0.01011(11) | -0.00083(8) | 0.00061(8) | 0.00014(9) |
| | neutron | 0.00410(30) | 0.00520(30) | 0.00650(30) | -0.00010(20) | 0.00060(30) | 0.00030(30) |
| | TAAM | 0.00858(7) | 0.00916(8) | 0.00999(8) | -0.00077(6) | 0.00057(6) | -0.00022(6) |
| | MM_iso | 0.00887(8) | 0.00952(8) | 0.01042(8) | -0.00087(6) | 0.00066(6) | -0.00021(6) |
| | MM_shade | 0.00884(7) | 0.00950(8) | 0.01041(8) | -0.00088(6) | 0.00064(6) | -0.00020(6) |
| | HAR_iso/HAR_shade | 0.00835(7) | 0.00894(8) | 0.00954(8) | -0.00074(6) | 0.00049(6) | -0.00018(6) |
| | HAR_aniso | 0.00833(7) | 0.00893(8) | 0.00959(8) | -0.00074(6) | 0.00048(6) | -0.00016(6) |
| C8 | IAM | 0.00748(9) | 0.01010(11) | 0.00926(10) | -0.00004(8) | 0.00114(8) | -0.00047(9) |
| | neutron | 0.00460(30) | 0.00560(30) | 0.00430(30) | 0.00040(20) | 0.00110(30) | -0.00030(20) |
| | TAAM | 0.00767(7) | 0.01016(8) | 0.00826(8) | 0.00032(6) | 0.00106(5) | -0.00081(6) |
| | MM_iso | 0.00795(6) | 0.01053(7) | 0.00858(8) | 0.00037(6) | 0.00107(5) | -0.00082(6) |
| | MM_shade | 0.00794(6) | 0.01054(7) | 0.00858(8) | 0.00037(6) | 0.00107(5) | -0.00083(6) |
| | HAR_iso/HAR_shade | 0.00734(6) | 0.00977(8) | 0.00804(8) | 0.00020(6) | 0.00105(5) | -0.00087(6) |
| | HAR_aniso | 0.00734(7) | 0.00975(8) | 0.00804(8) | 0.00020(6) | 0.00103(6) | -0.00088(6) |
| C9 | IAM | 0.01089(11) | 0.01388(12) | 0.00905(11) | 0.00255(10) | 0.00071(9) | -0.00159(10) |
| | neutron | 0.00680(40) | 0.00750(30) | 0.00470(30) | 0.00170(30) | 0.00050(30) | -0.00060(30) |
| | TAAM | 0.01099(8) | 0.01394(9) | 0.00872(8) | 0.00278(7) | 0.00054(6) | -0.00179(7) |
| | MM_iso | 0.01137(8) | 0.01423(9) | 0.00911(9) | 0.00277(8) | 0.00055(6) | -0.00176(7) |
| | MM_shade | 0.01135(8) | 0.01421(9) | 0.00910(9) | 0.00275(8) | 0.00053(6) | -0.00174(7) |
| | HAR_iso/HAR_shade | 0.01081(8) | 0.01369(9) | 0.00832(8) | 0.00277(7) | 0.00050(6) | -0.00176(7) |
| | HAR_aniso | 0.01078(8) | 0.01370(9) | 0.00838(8) | 0.00276(7) | 0.00053(6) | -0.00172(7) |
| C10 | IAM | 0.01267(13) | 0.01774(15) | 0.00874(12) | 0.00323(11) | 0.00105(10) | 0.00022(11) |
| | neutron | 0.00720(30) | 0.00890(30) | 0.00500(30) | 0.00250(30) | 0.00100(30) | 0.00050(30) |
| | TAAM | 0.01269(9) | 0.01721(11) | 0.00871(9) | 0.00371(8) | 0.00068(7) | -0.00007(7) |
| | MM_iso | 0.01292(9) | 0.01738(11) | 0.00933(10) | 0.00373(9) | 0.00066(7) | -0.00003(8) |
| | MM_shade | 0.01289(9) | 0.01734(11) | 0.00934(10) | 0.00374(8) | 0.00068(7) | -0.00002(8) |
| | HAR_iso/HAR_shade | 0.01242(8) | 0.0170(1) | 0.00842(9) | 0.00363(7) | 0.00063(7) | -0.00010(7) |
| | HAR_aniso | 0.01241(9) | 0.0171(1) | 0.00837(9) | 0.00363(8) | 0.00062(7) | -0.00013(8) |
| C11 | IAM | 0.01073(11) | 0.01518(13) | 0.01123(12) | 0.00286(10) | 0.00109(9) | 0.00157(11) |
| | neutron | 0.00580(30) | 0.00850(30) | 0.00600(30) | 0.00250(30) | 0.00130(30) | 0.00090(30) |
| | TAAM | 0.01076(8) | 0.01482(10) | 0.01142(9) | 0.00310(7) | 0.00125(6) | 0.00154(7) |
| | MM_iso | 0.01109(8) | 0.01517(10) | 0.01189(10) | 0.00315(8) | 0.00132(7) | 0.00156(8) |
| | MM_shade | 0.01106(8) | 0.01516(9) | 0.01187(10) | 0.00313(8) | 0.00133(7) | 0.00154(8) |
| | HAR_iso/HAR_shade | 0.01054(8) | 0.0147(1) | 0.01095(9) | 0.00310(7) | 0.00121(6) | 0.00161(8) |
| | HAR_aniso | 0.01051(8) | 0.0147(1) | 0.01098(9) | 0.00308(7) | 0.00125(7) | 0.00159(8) |

Table 9. *Values of ADPs of non-hydrogen atoms for various refinement strategies (IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_shade and HAR_aniso). The ADPs are expressed in the crystal coordinate system.*

| Model | | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|-------|-------------------|-------------|-------------|-------------|-------------|-------------|--------------|
| C12 | IAM | 0.01167(12) | 0.01475(13) | 0.01300(12) | 0.00401(11) | 0.00268(10) | -0.00040(11) |
| | neutron | 0.00750(30) | 0.00830(30) | 0.00620(30) | 0.00280(30) | 0.00110(30) | 0.00000(30) |
| | TAAM | 0.01155(8) | 0.01461(10) | 0.01184(9) | 0.00444(7) | 0.00240(7) | -0.00021(8) |
| | MM_iso | 0.01186(8) | 0.01501(9) | 0.01219(10) | 0.00442(8) | 0.00245(7) | -0.00019(8) |
| | MM_shade | 0.01185(8) | 0.01501(9) | 0.01213(10) | 0.00443(8) | 0.00242(7) | -0.00017(8) |
| | HAR_iso/HAR_shade | 0.01130(8) | 0.0143(1) | 0.01159(9) | 0.00439(7) | 0.00238(7) | -0.00025(8) |
| | HAR_aniso | 0.01135(8) | 0.0142(1) | 0.01157(9) | 0.00438(7) | 0.00232(7) | -0.00026(8) |
| C13 | IAM | 0.01073(11) | 0.01362(12) | 0.00956(11) | 0.00232(10) | 0.00289(9) | -0.00082(10) |
| | neutron | 0.00670(30) | 0.00760(30) | 0.00500(30) | 0.00190(30) | 0.00240(30) | -0.00020(20) |
| | TAAM | 0.01055(8) | 0.01367(9) | 0.00924(8) | 0.00295(7) | 0.00214(6) | -0.00077(7) |
| | MM_iso | 0.01095(8) | 0.01405(9) | 0.00957(9) | 0.00301(8) | 0.00229(6) | -0.00078(7) |
| | MM_shade | 0.01090(8) | 0.01402(9) | 0.00958(9) | 0.00300(8) | 0.00229(6) | -0.00076(7) |
| | HAR_iso/HAR_shade | 0.01035(7) | 0.01349(9) | 0.00875(8) | 0.00289(7) | 0.00215(6) | -0.00074(7) |
| | HAR_aniso | 0.01036(8) | 0.01342(9) | 0.00883(9) | 0.00291(7) | 0.00208(6) | -0.00076(7) |

Table 10. *Values of IDPs of hydrogen atoms for various refinement strategies (IAM, TAAM, MM_iso and HAR_iso). The IDPs are expressed in the crystal coordinate system.*

| Method | H1 | H2 | H3 | H41 | H42 | H43 | H5 | H6 |
|---------|----------|----------|----------|----------|----------|----------|----------|----------|
| IAM | 0.042(3) | 0.016(2) | 0.022(2) | 0.021(2) | 0.017(2) | 0.026(3) | 0.045(4) | 0.004(2) |
| TAAM | 0.034(4) | 0.026(2) | 0.028(2) | 0.028(2) | 0.031(3) | 0.029(2) | 0.033(3) | 0.024(2) |
| MM_iso | 0.026(6) | 0.024(3) | 0.028(4) | 0.025(3) | 0.023(3) | 0.020(3) | 0.031(5) | 0.017(4) |
| HAR_iso | 0.029(3) | 0.027(2) | 0.027(2) | 0.031(2) | 0.029(2) | 0.026(2) | 0.026(2) | 0.016(1) |

| Method | H71 | H72 | H9 | H10 | H11 | H12 | H13 |
|---------|----------|----------|----------|----------|----------|----------|----------|
| IAM | 0.026(3) | 0.010(2) | 0.023(2) | 0.025(3) | 0.016(2) | 0.023(3) | 0.022(2) |
| TAAM | 0.031(2) | 0.024(2) | 0.035(3) | 0.029(2) | 0.030(2) | 0.027(2) | 0.025(2) |
| MM_iso | 0.019(3) | 0.022(3) | 0.021(3) | 0.023(3) | 0.021(3) | 0.023(3) | 0.026(3) |
| HAR_iso | 0.023(2) | 0.022(2) | 0.032(2) | 0.030(2) | 0.028(2) | 0.031(2) | 0.026(2) |

Table 11. *Geometry of hydrogen bonds present in the structure resulting from various refinement methods: IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_aniso and HAR_shade. Units of distances and angles: Å, deg.*

| Bond | Method | D - H | H···A | D···A | D - H···A |
|-------------------|-----------|-----------|-----------|------------|------------|
| O1-H1···O2 | IAM | 1.142(19) | 1.270(19) | 2.4122(7) | 178.4(17) |
| SYMM | neutron | 1.209(3) | 1.208(3) | 2.4163(17) | 176.0(3) |
| - | TAAM | 1.218(17) | 1.194(16) | 2.4110(3) | 178.3(14) |
| | MM_iso | 1.177(39) | 1.239(39) | 2.4103(5) | 171.5(37) |
| | MM_shade | 1.166(41) | 1.253(41) | 2.4102(5) | 170.6(37) |
| | HAR_iso | 1.188(17) | 1.223(17) | 2.4106(3) | 178.4(15) |
| | HAR_aniso | 1.205(18) | 1.207(18) | 2.4113(3) | 177.3(12) |
| | HAR_shade | 1.192(15) | 1.219(15) | 2.4106(3) | 177.9(14) |
| O5-H5···O3 | IAM | 0.842(15) | 1.683(15) | 2.5214(7) | 174.2(15) |
| SYMM | neutron | 1.053(3) | 1.475(3) | 2.5231(15) | 173.3(3) |
| - | TAAM | 1.018(3) | 1.502(3) | 2.5196(3) | 178.30(11) |
| | MM_iso | 1.018(4) | 1.503(4) | 2.5207(5) | 178.47(5) |
| | MM_shade | 1.018(4) | 1.503(4) | 2.5207(5) | 178.40(10) |
| | HAR_iso | 1.025(15) | 1.499(15) | 2.5206(3) | 174.4(11) |
| | HAR_aniso | 1.014(17) | 1.508(17) | 2.5207(3) | 176.4(13) |
| | HAR_shade | 1.027(13) | 1.497(13) | 2.5205(3) | 174.1(10) |
| N1-H41···O4 | IAM | 0.860(11) | 2.132(11) | 2.9830(8) | 170.2(10) |
| SYMM | neutron | 1.038(3) | 1.952(3) | 2.9723(15) | 166.9(3) |
| [x, y + 1, z + 1] | TAAM | 1.036(3) | 1.951(4) | 2.9634(4) | 173.50(12) |
| | MM_iso | 1.036(4) | 1.949(4) | 2.9663(4) | 173.19(14) |
| | MM_shade | 1.036(4) | 1.949(4) | 2.9662(4) | 173.35(14) |
| | HAR_iso | 1.024(14) | 1.968(14) | 2.9822(3) | 170.0(11) |
| | HAR_aniso | 1.037(13) | 1.957(13) | 2.9820(3) | 169.1(10) |
| | HAR_shade | 1.022(10) | 1.971(10) | 2.9821(3) | 169.5(9) |
| N1-H41···O2 | IAM | 0.860(11) | 2.344(11) | 2.9765(7) | 130.7(9) |
| SYMM | neutron | 1.038(3) | 2.218(3) | 2.9763(15) | 128.4(2) |
| [x, y + 1, z + 1] | TAAM | 1.036(3) | 2.2727(4) | 2.9779(3) | 123.9(2) |
| | MM_iso | 1.036(4) | 2.2714(6) | 2.9799(4) | 124.2(2) |
| | MM_shade | 1.036(4) | 2.2731(6) | 2.9800(4) | 124.1(2) |
| | HAR_iso | 1.024(14) | 2.251(12) | 2.9784(3) | 126.7(10) |
| | HAR_aniso | 1.037(13) | 2.236(12) | 2.9782(3) | 127.0(11) |
| | HAR_shade | 1.022(10) | 2.246(10) | 2.9784(3) | 127.3(8) |
| N1-H42···O4 | IAM | 0.828(11) | 2.164(11) | 2.9628(8) | 161.9(11) |
| SYMM | neutron | 1.041(3) | 1.976(3) | 2.9803(15) | 161.2(3) |
| [x, y, z + 1] | TAAM | 1.036(3) | 1.975(4) | 2.9822(3) | 158.57(12) |
| | MM_iso | 1.036(4) | 1.976(4) | 2.9802(5) | 158.89(14) |
| | MM_shade | 1.036(4) | 1.977(4) | 2.9803(5) | 158.71(14) |
| | HAR_iso | 0.992(12) | 2.002(12) | 2.9630(4) | 162.4(10) |
| | HAR_aniso | 0.999(13) | 1.990(14) | 2.9632(4) | 164.2(11) |
| | HAR_shade | 0.996(10) | 2.001(10) | 2.9630(3) | 161.5(9) |

Table 12. *Geometry of hydrogen bonds present in the structure resulting from various refinement methods: IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_aniso and HAR_shade. Units of distances and angles: Å, deg.*

| Bond | Method | D - H | H ··· A | D ··· A | D - H ··· A |
|-----------------------|-----------|------------|------------|------------|-------------|
| N1-H43 ··· O3 | IAM | 0.870(12) | 2.205(12) | 2.9963(7) | 151.1(11) |
| SYMM | neutron | 1.032(2) | 2.100(3) | 2.9978(15) | 144.0(2) |
| [$-x, y + 1/2, -z$] | TAAM | 1.0360(9) | 2.1151(5) | 2.9962(3) | 141.51(5) |
| | MM_iso | 1.0360(11) | 2.1159(5) | 2.9968(3) | 141.48(6) |
| | MM_shade | 1.0359(11) | 2.1158(6) | 2.9970(3) | 141.53(6) |
| | HAR_iso | 1.040(10) | 2.080(10) | 2.9960(3) | 145.6(9) |
| | HAR_aniso | 1.022(10) | 2.129(9) | 2.9960(3) | 141.4(9) |
| | HAR_shade | 1.044(9) | 2.080(9) | 2.9961(3) | 145.1(8) |
| N1-H43 ··· O6 | IAM | 0.870(12) | 2.416(12) | 2.9798(7) | 122.9(10) |
| SYMM | neutron | 1.032(2) | 2.276(3) | 2.9753(15) | 123.7(2) |
| [$-x, y + 1/2, -z$] | TAAM | 1.0360(9) | 2.2866(16) | 2.9804(3) | 87.25(13) |
| | MM_iso | 1.0360(11) | 2.2909(18) | 2.9799(3) | 87.54(15) |
| | MM_shade | 1.0359(11) | 2.2882(18) | 2.9794(3) | 122.8(2) |
| | HAR_iso | 1.040(10) | 2.302(11) | 2.9803(3) | 121.5(8) |
| | HAR_aniso | 1.022(10) | 2.277(12) | 2.9801(3) | 124.7(9) |
| | HAR_shade | 1.044(9) | 2.296(10) | 2.9804(3) | 121.8(8) |

Table 13. *Non-hydrogen bond lengths resulting from various refinement methods: IAM, neutron, TAAM, MM_iso, MM_shade, HAR_iso, HAR_aniso, HAR_shade.*

| Bond | IAM | neutron | TAAM | MM_iso | MM_shade | HAR_iso | HAR_aniso | HAR_shade |
|---------|-----------|------------|-----------|-----------|-----------|-----------|-----------|-----------|
| O1-C1 | 1.2736(5) | 1.2748(13) | 1.2726(3) | 1.2730(4) | 1.2729(4) | 1.2722(2) | 1.2721(2) | 1.2723(2) |
| C1-C2 | 1.4931(6) | 1.4981(14) | 1.4926(4) | 1.4928(4) | 1.4929(4) | 1.4928(2) | 1.4933(2) | 1.4928(2) |
| C1-O3 | 1.2532(5) | 1.2507(14) | 1.2508(3) | 1.2500(4) | 1.2501(4) | 1.2507(2) | 1.2501(2) | 1.2505(2) |
| C2-C3 | 1.3435(6) | 1.3427(14) | 1.3451(4) | 1.3447(4) | 1.3450(4) | 1.3446(2) | 1.3445(2) | 1.3448(2) |
| C4-O2 | 1.2884(6) | 1.2891(14) | 1.2872(4) | 1.2892(5) | 1.2889(5) | 1.2878(2) | 1.2876(2) | 1.2878(2) |
| C3-C4 | 1.4907(6) | 1.4926(13) | 1.4913(4) | 1.4916(4) | 1.4918(4) | 1.4919(2) | 1.4916(2) | 1.4917(2) |
| C4-O4 | 1.2412(5) | 1.2450(15) | 1.2406(3) | 1.2400(4) | 1.2399(4) | 1.2401(2) | 1.2404(2) | 1.2400(2) |
| O5-C5 | 1.3098(5) | 1.3051(17) | 1.3076(3) | 1.3076(4) | 1.3076(4) | 1.3078(2) | 1.3077(2) | 1.3078(2) |
| C5-O6 | 1.2186(5) | 1.2195(15) | 1.2189(3) | 1.2197(4) | 1.2194(4) | 1.2181(2) | 1.2181(2) | 1.2181(2) |
| C6-N1 | 1.4883(6) | 1.4890(12) | 1.4871(3) | 1.4866(4) | 1.4866(4) | 1.4880(3) | 1.4880(3) | 1.4880(3) |
| C5-C6 | 1.5153(6) | 1.5195(13) | 1.5152(3) | 1.5150(4) | 1.5149(4) | 1.5153(2) | 1.5152(2) | 1.5151(2) |
| C6-C7 | 1.5315(6) | 1.5304(13) | 1.5317(3) | 1.5308(4) | 1.5310(4) | 1.5314(2) | 1.5315(2) | 1.5313(2) |
| C7-C8 | 1.5071(6) | 1.5089(13) | 1.5081(4) | 1.5077(4) | 1.5078(4) | 1.5083(2) | 1.5084(2) | 1.5082(2) |
| C8-C9 | 1.3989(6) | 1.3971(13) | 1.3973(4) | 1.3976(4) | 1.3976(4) | 1.3973(2) | 1.3970(2) | 1.3972(2) |
| C8-C13 | 1.3967(6) | 1.3999(14) | 1.3983(4) | 1.3981(4) | 1.3982(4) | 1.3979(2) | 1.3982(2) | 1.3978(2) |
| C9-C10 | 1.3912(7) | 1.3973(13) | 1.3919(4) | 1.3920(4) | 1.3920(4) | 1.3922(2) | 1.3919(2) | 1.3919(2) |
| C10-C11 | 1.3924(7) | 1.3933(15) | 1.3921(4) | 1.3919(4) | 1.3920(4) | 1.3921(2) | 1.3925(2) | 1.3919(2) |
| C11-C12 | 1.3934(7) | 1.3940(14) | 1.3931(4) | 1.3929(4) | 1.3931(4) | 1.3922(2) | 1.3921(2) | 1.3922(2) |
| C12-C13 | 1.3926(7) | 1.3962(13) | 1.3945(4) | 1.3942(4) | 1.3943(4) | 1.3943(2) | 1.3943(2) | 1.3943(2) |